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A SURVEY OF THE THERMODYNAMICS OF HAN-BASED LIQUID GUN PROPELLANTS (U)

Eli Freedman

December 1980

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (meg)

(U) There are many reasons for thinking that the first liquid gun propellant to achieve type-classification will be a mixture of hydroxylammonium nitrate (HAN), water, and a miscible organic fuel. A survey has been made of the impetus produced by such systems with more than twenty fuels. The results are found to depend strongly on the amount of water present in the mixture. The range of impetuses computed is surprisingly small, all of them lying within ten percent of either side of the mean. The ratio of oxidizer to fuel has only a slight

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20. Abstract (Cont'd):

effect on the results, although a flat maximum occurs on the fuel-rich side. The effect of non-ideal gas corrections on the results is noticeable, but still within the precision of available closed-bomb data. Significant advances in computations of this type require improved closed-bomb data.

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(U) TABLE OF CONTENTS

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(U) LIST OF TABLES

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I. (U) INTRODUCTION

(U) The search for acceptable liquid gun propellants (LGPs) has been going on for decades. Since most of the exposure of troops to LGPs will occur in training exercises, not in battle, the recent heightened awareness of the need for protection of personnel from toxic or corrosive materials has sharply reduced the variety of suitable materials for consideration. Thus, although hydrazine and some of its derivatives have shown good, even excellent, ballistic performance in tests, they must be regarded as unacceptable. Likewise, organic nitrates (e.g., propylene glycol dinitrate) are much too active physiologically to merit further consideration.

(C) These toxicological arguments are admittedly a posteriori, because a class of LGPs that have low vapor pressures and are non-corrosive has been known for some time. These LGPs contain hydroxylammonium nitrate (HAN) as an oxidizer, an organic fuel (a suitably chosen compound of C, H, O, and perhaps N), and water. Table 1 lists the compositions of some of these propellants. The most important member of this class is NOS-365, originally developed by the Navy[1] as a torpedo fuel to replace OTTO-II, which contains an organic nitrate.

(C) NOS-365 contains isopropylammonium nitrate (IPAN) as its fuel, and 20% by weight of water. (Henceforth, all percentages will be understood to be weight-percents, and will not be spelled out.) The HAN/IPAN mixture forms a subclass of HAN-based LGPs, each differing mainly in the percentage of water present.

(C) The ultimate test of any propellant, solid, liquid, or gas, is its behavior in a gun. Since gun firings are expensive, the more that can be learned by computer studies and small-scale laboratory testing, the better. The present paper is devoted entirely to thermodynamic computer studies. It has three aims: To see if IPAN is thermodynamically the best fuel; to consider the effect of the amount of water on computed performance; and to consider the effect of oxidizer to fuel ratio (O/F) on performance.

II. (U) METHODOLOGY

(U) The BLAKE[2] thermodynamics program was used throughout this work. Although it is slower than other codes, and has a comparatively limited library, it has the great advantage of being able to take into account

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[1] *(U) "Liquid Monopropellant Gun Exploratory Development Program," IHTR 405, 30 Aug 74 (Confidential report).*

[2] *(U) E. Freedman, "BLAKE -- A Thermochemical Code Based on TIGER," Paper 10 in "Proceedings of the International Symposium on Ballistics", Dover, NJ, 1973.*

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TABLE 1. (U) COMPOSITION OF AQUEOUS MONOPROPELLANTS

	NOS-283		NOS-365		NOS-411	
COMPONENT	WT %	MOBILITY	WT %	MOBILITY	WT %	MOBILITY
HAN	56.9	8.08	60.7	8.83	64.5	9.47
IPAN	18.1	2.02	19.3	2.21	20.5	2.37
H_2O	25.0	18.90	20.0	15.50	15.0	11.80
Density (g/cm^3)	1.363		1.396		1.410	

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TABLE 2. (U) ENTHALPIES OF REACTION FOR ORGANIC AMINES WITH NITRIC ACID

AMINE	$\Delta H°$ (Reaction) joule per mole of amine
Methylamine	-132.8
Ethylamine	-118.5
Trimethylamine	- 89.7
Triethylamine	-105.1
Dimethylamine	-114.8

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non-ideal gas effects. The code's application to LGPs containing HAN and water, while good, nevertheless leaves some room for improvement. The method for treating non-ideal gas effects in BLAKE applies strictly only to spherical nonpolar molecules, and is thus incorrect for water. This error is partially compensated by using appropriately adjusted values of the Lennard-Jones force constants from which the corrections for non-ideality are derived. In the case of LGPs where more than half of the product gases are water, the compensation is insufficient.

(U) The effect of this error is not so great as to invalidate either the comparisons made, or any of the conclusions reached.

(U) The code could readily be improved by incorporating the Stockmayer potential for water and ammonia, the two principal "offenders." But until closed-bomb values for LGPs are available, such a modification remains an unnecessary frill.

(U) A much more significant input parameter is the enthalpy of formation of the ingredients, and much effort was exerted to obtain the best available data in the literature. Wherever possible, data were taken from Cox and Pilcher's monumental compilation[3]. Some additional important values were located in Domalski's survey[4].

(U) The only significant compounds for which data were lacking were some organic ammonium nitrates. Domalski[4] gives enthalpies of formation for five of them, the salts of methyl, dimethyl, trimethyl, diethyl, and triethyl amines. Enthalpies of formation for all of the amines are in Cox and Pilcher[3]. Combining the data from these two sources, we get the enthalpies of reaction for the formation of the salts from their amines as shown in Table 2.

(C) The average enthalpy of reaction for these five salts is -112 ± 22 J. Excluding methylammonium nitrate changes the average to -107 ± 7 J. (This exclusion is reasonable because the first member of a homologous series often differs significantly from the higher members.) Ultimately, the value of -117 J, the value for ethyl, was chosen for both n-propyl and isopropyl ammonium nitrates, leading to the enthalpies of formation shown in Table 3.

(U) The enthalpy of formation of HAN used for all of these calculations was -366.5 J/mol[5].

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[3] *(U) J.D. Cox and G. Pilcher, "Thermochemistry of Organic and Organometallic Compounds", Academic Press, London, 1970.*

[4] *(U) E.S. Domalski, J. Physical Chemical Ref. Data, Vol. 1, p. 221, 1972.*

[5] *(U) "Selected Values of Chemical Thermodynamic Properties", NBS Technical Note 270-3, 1968.*

TABLE 3. (U) SUMMARY OF THERMODYNAMIC COMPUTATIONS ON HAN-BASED LIQUID GUN PROPELLANTS

(Water = 20% by Weight; Loading Density = 0.2 g/cm^3; L-J sigma = 10^{-6} m)

Fuel	C/H/N/O	$-\Delta H^\circ_f$ (J/mole)	O/F	I(J/g)	Notes
Ethyleneimine Nitrate	2/7/2/3	211.00	2.25	976.9	1
Trimethylammonium Nitrate (TMAN)	3/10/2/3	310.0	4.0	955.5	1
Tetramethylammonium Nitrate	4/12/2/3	344.0	5.5	950.4	1
Triethylammonium Nitrate	6/16/2/3	413.8	8.5	942.7	1
11-n-decylhenicosane	31/64/0/0	848.1	47.0	942.4	2
Tri-Octylammonium Nitrate	24/25/2/3	883.	35.5	942.2	1
Dimethylammonium Nitrate	2/8/2/3	346.9	2.5	937.1	3
Triethanolammonium Nitrate	6/16/2/6	728.	7.0	934.8	4
n-butylammonium Nitrate	4/12/2/3	431.	5.5	932.5	1
Isopropylammonium Nitrate (IPAN)	3/10/2/3	403.3	4.0	930.0	3
n-propylammonium Nitrate	3/10/2/3	404.6	4.0	929.7	3
2, 2-diethoxypropane	7/16/0/2	539.02	10.0	927.3	3
Dodecanoic Acid	12/26/0/2	774.58	17.5	925.4	2
Ethylammonium Nitrate	2/8/2/3	366.9	2.5	924.9	1
1, 4-dioxane	4/8/0/2	353.42	5.0	920.7	2
Tribenzylammonium Nitrate	18/16/2/3	155.	20.5	917.7	3
Pyridinium Nitrate	5/6/2/3	197.	5.0	916.6	3
Anilinium Nitrate	6/8/2/3	271.5	6.5	913.2	3
1, 3-dioxane	4/8/0/2	385.81	5.0	912.8	2
Methylammonium Nitrate	1/6/2/3	354.4	1.0	911.4	1
Ethylammonium Nitrate	2/8/2/4	517.1	2.0	897.8	4
8-hydroxyquinoline Nitrate	9/8/2/4	385.	9.0	895.3	3
Ethylenediammonium Nitrate	2/10/4/6	653.5	1.5	892.6	1
Ethylene Glycol	2/6/0/2	454.93	2.5	868.8	2
Triacetin	9/14/0/6	1331.	9.5	847.5	2
OTTO-II	-/-/-/-	---	---	866.7	-

Notes: 1 - ΔH°_f *from Ref. 4.*
2 - ΔH°_f *from Ref. 3.*
3 - ΔH°_f *for nitrate salt estimated by this writer.*
4 - ΔH°_f *for both amine and its nitrate estimated by this writer.*

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(C) Another source of uncertainty in the enthalpy of formation data concerns the physical state of the individual ingredients in the final mixture. Propellant thermodynamic calculations are almost always made with the assumption that the enthalpy of formation of the final propellant is the sum of the enthalpies of formation of the ingredients, each weighted by its mole fractions. This procedure neglects all energies of interaction, and is a reasonable assumption for solid propellants. It may not be quite so reasonable for LGPs containing water, since heats of hydration are occasionally large. In the present case, the heat of solution of HAN is -22 J/mol for a 1 M solution[5]. The heat of solution decreases with increasing concentration; since NOS-365 contains 8.8 M HAN, it appeared that the value for the crystalline state was more appropriate than that for the solvate substance. (It is worth noting that in NOS-365 there are only two water molecules for each molecule of HAN.) The same assumption was made for all of the fuels, there being no data on heats of solution in any case. The necessary corrections can be made at once when more or better data become available, but the changes will not be significant.

(C) As will be shown later, the amount of water has a large effect on the results. This brings up a problem of a different sort. Most chemists working with solutions prefer to specify concentrations in molarities (i.e., the number of moles in 1000 cm^3 of solution), since they can then use volumes, which are readily and conveniently measured, to determine the amounts of reactants. Unfortunately, molarities change with density. Different fuels have different densities, and there is no general method known for computing the density of a solution from its ingredients other than by measurement after formulation. How then should the comparison between fuel A and fuel B be made? For example, NOS-365 can be specified in two different, although equivalent, ways either containing 60.70% HAN, 19.30% IPAN, and 20.00% water, or an containing 8.85 M HAN, 2.02 M IPAN, and 15.54 M water. Suppose one wants to compare the calculated performance of NOS-365 with that of a propellant containing a different fuel of the same empirical formula ($C_3H_{10}N_2O_3$), but with a density of 1.26 g/cm^3 (compared to 1.4 g/cm^3 for NOS-365). The percentage composition of this latter propellant is identical with that of NOS-365, but its molar composition is markedly different: 7.96 M HAN, 1.99 M fuel, and 13.99 M water. If this latter propellant is formulated to have the same molarities as NOS-365, then the percentage of water, for example, becomes 11.12%. This effect is evidently not trivial. In the belief that comparisons based on weight are more meaningful, they have been used consistently. This point should be considered an assumption, not a proven fact.

(U) All of the calculations were carried out uniformly for a loading density of 0.2 g/cm^3. One of the advantages of LGPs is that they have high intrinsic densities, but there is no reason for making thermodynamic computations at such densities because shot start always occurs well before all-burnt. The effective loading density at shot start is likely to be greater than 0.2 g/cm^3, but this number is a convenient

one for comparisons. It is large enough to shown non-ideal gas effects, but not so large for them to have disproportionate effects on the results.

(U) The applications of LGPs in the field will likely be in volume-limited systems, which would make the volumetric impetus (impetus multiplied by density) the more useful quantity for comparison. Results obtained in this laboratory have shown that the densities of many HAN-based LPs can be estimated moderately well by assuming that specific volumes (the reciprocal of the densities) are additive. Thus, the density d, of a particular solution is

$$1/d = \Sigma\ w(i)/d(i)$$

where w(i) is the weight fraction of component i, of density d(i). This equation works well provided the quantities d(i) for the individual components are chosen to fit some experimental points, rather than using handbook values. This is especially important for materials like HAN, which is a solid in its pure state. This additional step has not been included in the results reported here.

(U) To summarize: All of the results to be presented are for a specified weight-percent of water (usually 20%), for a specified oxidizer to fuel ratio (usually stoichiometric), and always for a loading density of 0.2 g/cm^3.

III. (U) RESULTS: THE EFFECT OF STRUCTURE

(C) Table 3 presents the computed impetuses for 25 fuels, and also for OTTO-II. The most striking aspects of this table is how small the range of impetuses are. The distribution is plotted in Figure 1. Even though the fuels range from hydrocarbons to organic ammonium nitrates, from short chains to long chains and to rings, from aliphatic to aromatic to heterocyclic, the entire range of impetuses is only 847 to 977 J/g; the average value is 948. The dominant property determining the results is the enthalpy of formation, but the empirical formula does have some effect.

(C) The separation of the effect of the numbers of C, H, N, and O atoms on the calculated impetus from the effect of enthalpy is not obvious. An additional complication is the fact that the computations are carried out for stoichiometric mixtures with HAN. For a stoichiometric mixture of HAN ($H_4N_2O_4$) with a compound $C_xH_yO_z$ (the number of nitrogens is irrelevant), the ratio of oxidizer to fuel at stoichiometry is given by

$$O/F = x + (y/4) - (z/2)$$

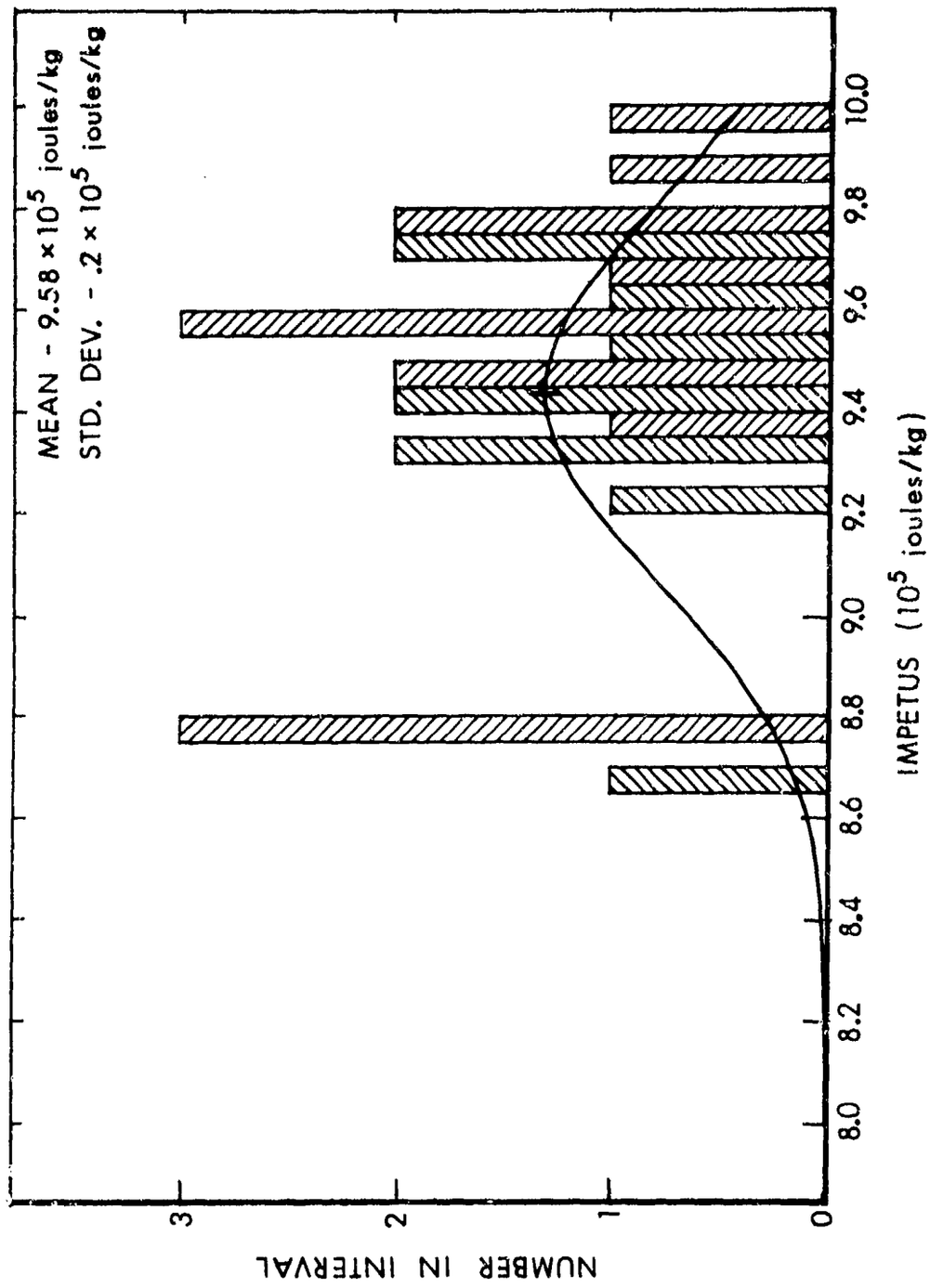

Figure 1. (U) Distribution of Computed Impetus of Liquid Propellants.

Changing x or y or z in general changes O/F (except for accidental cancellations). The overall result is a system with too many variables to permit the isolation of significant factors.

(U) M. Summerfield[6] suggested that one way of looking for trends would be to consider the effects of a homologous series of the same family of compounds. For example, lengthening a carbon chain by adding one methylene group (--CH_2--) makes the enthalpy of formation about 21 J/mol more negative. Assuming that the enthalpy of formation of a C-4 ammonium nitrate is about -350 J/mol, one sees that the enthalpy of formation of an organic ammonium nitrate with n carbons is

$$\Delta H = -350-21(n-4)$$

For alcohols, the corresponding equation is

$$\Delta H = -305-21(n-4).$$

Table 4 shows the results of the calculation for the impetus as a function of n at stoichiometry and 20% water. The impetus increases with increasing chain length, but the rate of increase declines as the chain gets longer. The limiting value depends almost entirely on the fuel's enthalpy of formation, and hardly at all on its formula.

(C) We conclude that, based on all of the calculations performed up to now, the impetus of a liquid gun propellant composed of HAN in stoichiometric proportions with almost any fuel, and containing 20% by weight water, will fall in the range of 900 to 1000 J/g.

(U) This conclusion will not hold for molecules with sufficiently small negative enthalpies or positive enthalpies of any size. Empirically, however, it is known that such compounds are prone to decompose at or near ambient temperatures and thus, will not be suitable for LGPs. Likewise, fuels with exceptionally large negative enthalpies will have impetuses below 0.9 kJ/g, but such propellants are not likely to be useful.

(U) One additional assumption implicit in the foregoing calculations that has not been mentioned yet is that all of the listed compounds can be made.[7] In fact, this is false for some of the listed organic ammonium nitrates. While there were some individual variations, attempts at synthesizing the organic ammonium nitrates with more than four carbons

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[6] *(U) M. Summerfield, private communication, May 77.*

[7] *(U) K.E. Travis, Interior Ballistics Division, BRL: Presentation given at the BRL Workshop on Liquid Propellants, May 77.*

TABLE 4. (U) EFFECT OF CHAIN LENGTH ON COMPUTED IMPETUS

(Conditions the same as for Table 3)

Class of Compound	No. of Carbons	$-\Delta H^\circ_f$ (J/mol)	Impetus (J/g)
Organic Ammonium Nitrate	4	350	973.67
$C_nH_{2n+4}N_2O_3$	6	392	973.73
	8	434	973.60
	10	476	973.76
Alcohol	4	305	956.65
$C_nH_{2n+2}O$	6	347	962.30
	8	389	965.16
	10	431	966.88
Hydroxy Acid	6	720	916.57
$C_nH_{2n+2}O_3$	7	753	923.88
	9	820	933.26
	11	895	938.32

generally resulted in the amines being oxidized rather than nitrated. Tetramethylammonium nitrate was readily synthesized, but a mixture of it with HAN refused to burn on a hot plate, whereas NOS-365 reacts immediately in such a test.

IV. (U) RESULTS: EFFECT OF WATER

(C) In striking contrast to the weak dependence of impetus on the nature of the fuel is the strong dependence of the calculated impetus on the amount of water in the mixture. This effect is shown in Figure 2 for two formulations, NOS-365 and 1776. (Formulation 1776 is similar to NOS-365, except for the substitution of trimethylammonium nitrate for IPAN; also, the percentages are a trifle different for reasons explained elsewhere[8]).

(C) The effect of adding water is almost exactly linear: a one percent increase in the water content causes a one percent decrease in the impetus. This effect arises from the low molecular weight of water (18.015) compared to that of any other ingredient in the propellant. A change of one gram of water in a hundred grams of propellant (1%) of average molecular weight, 100 means a change of about 5.4% in the number of molecules in the propellant, accounting for the disproportionate effect of water. A similar, although less dramatic, effect has been noted in solid propellants.

V. (U) RESULTS: EFFECT OF OXIDIZER TO FUEL RATIO

(U) All of the calculations of the section entitled, "Results: The Effect of Structure", were made for stoichiometric mixtures; that is, the amount of HAN in the propellant was just sufficient to oxidize all of the carbon to carbon dioxide and all of the hydrogen to water. It has long been known that rocket fuels achieve maximum specific impulse of O/F ratios smaller than stoichiometric (i.e., fuel-rich); the same question naturally arises for LGPs.

(U) The answer turns out to be the same as for rocket propellants. This is shown in Figure 3. With LGPs, however, the effect is quite weak, so that there is no compelling reason to attempt to formulate LGPs at the precise maximum impetus. For any particular fuel, the actual O/F ratio, and the even more important matter of the amount of water in the formulation, will be determined not by the impetus, but by the burning characteristics of the propellant.

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[8] *(U) E. Freedman and K. Travis, "Compositions, Nomenclature, and Densities of Aqueous Liquid Gun Propellants", Ballistic Research Laboratory Memorandum Report to be published.*

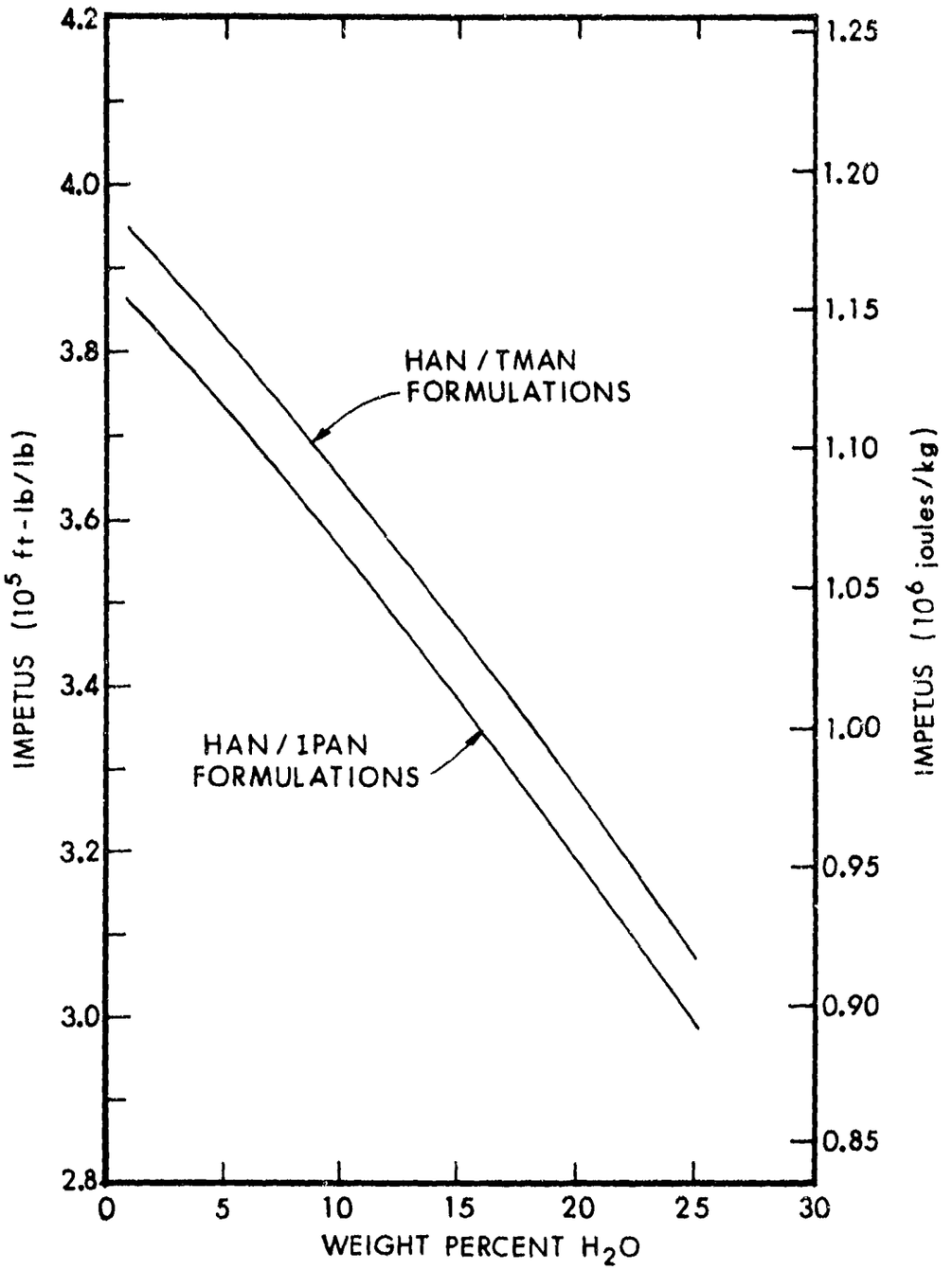

Figure 2. (U) Effect of H_2O on Impetus of Two Liquid Propellants.

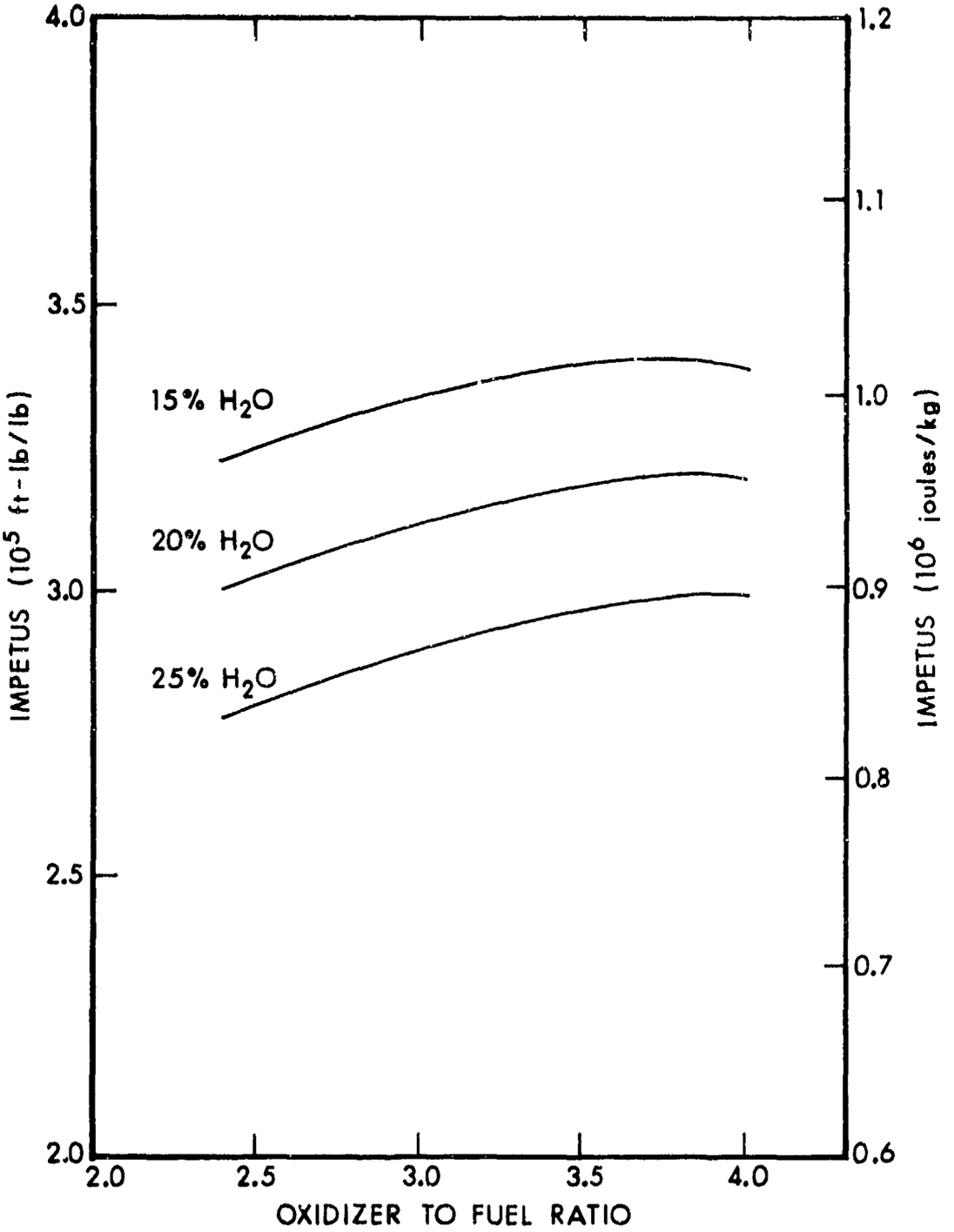

Figure 3. (U) Effect of Oxidizer/Fuel Ratio on Three Liquid Propellants.

VI. (U) RESULTS: BIPROPELLANTS

(U) BLAKE, in common with all other thermodynamic codes, assumes that all of the ingredients are mixed and burned homogeneously, regardless of the physical facts. (The discrepancy is especially noteworthy with solid small-arms propellants.) At first sight, then it might appear that bipropellants, whose components are immiscible with each other, could not be treated in the same way. Provided the combustion of the two components is complete, however, it is immaterial whether the initial starting materials were mixed or not. Table 5 summarizes calculations for a typical bipropellant, using decalin as the fuel and HAN as the oxidizer. Here, too, the strong dependence on the water content is noted. These results are shown graphically in Figure 4, where they are compared with NOS-365.

VII. (U) DISCUSSION AND CONCLUSIONS

(U) LP guns can be designed to use either monopropellants or bipropellants. Test firings in both bulk-loaded and regeneratively-injected LP guns have demonstrated high ballistic efficiencies. Regardless of which combination is used, thermodynamic calculations of the kind presented here will be generally valid, subject to assumptions already mentioned. Thus, the present results are not affected by the vigorous controversy that rages over the type of propellant, the gun design, and various other related matters.

(C) A glance at Table 3 shows that the class of HAN-based propellants does indeed offer considerable promise. Since the typical HAN-based propellant has a density of 1.2 g/cm^3 (or greater), these propellants possess even more spectacular volumetric impetuses.

(U) In conclusion: (1) the impetus of a HAN-based liquid gun propellant depends only weakly on the exact nature of the fuel; (2) it is a strong function of the amount of water present; (3) the impetus varies only weakly with the oxidizer to fuel ratio; (4) maximum impetus occurs with fuel-rich systems, but the effect is not worth consideration at this time; and (5) the volumetric impetus of HAN-based liquid propellants makes them even more attractive as LGP candidate systems.

(U) ACKNOWLEDGMENTS

(U) It is a pleasure to thank my colleagues, with whom all of the details of the report have been discussed at one time or another, especially Dr. Nathan Klein and Dr. Kenton E. Travis. Thanks are also due to Dr. Travis for his discussing with me his work on ingredient synthesis in advance of its publication. I also want to thank Mr. R.D. Anderson (Ignition and Combustion Branch) for advice on the UNIVAC, and Dr. Walter F. Morrison for technical assistance.

TABLE 5. (U) IMPETUSES OF SOME HAN-BASED BIPROPELLANTS

Fuel	O/F	Water (%)	Impetus (J/g)
Decalin	12/1	1	1187.5
Decalin	12/1	5	1142.4
Decalin	12/1	15	1019.6
Decalin	14/1	20	969.0

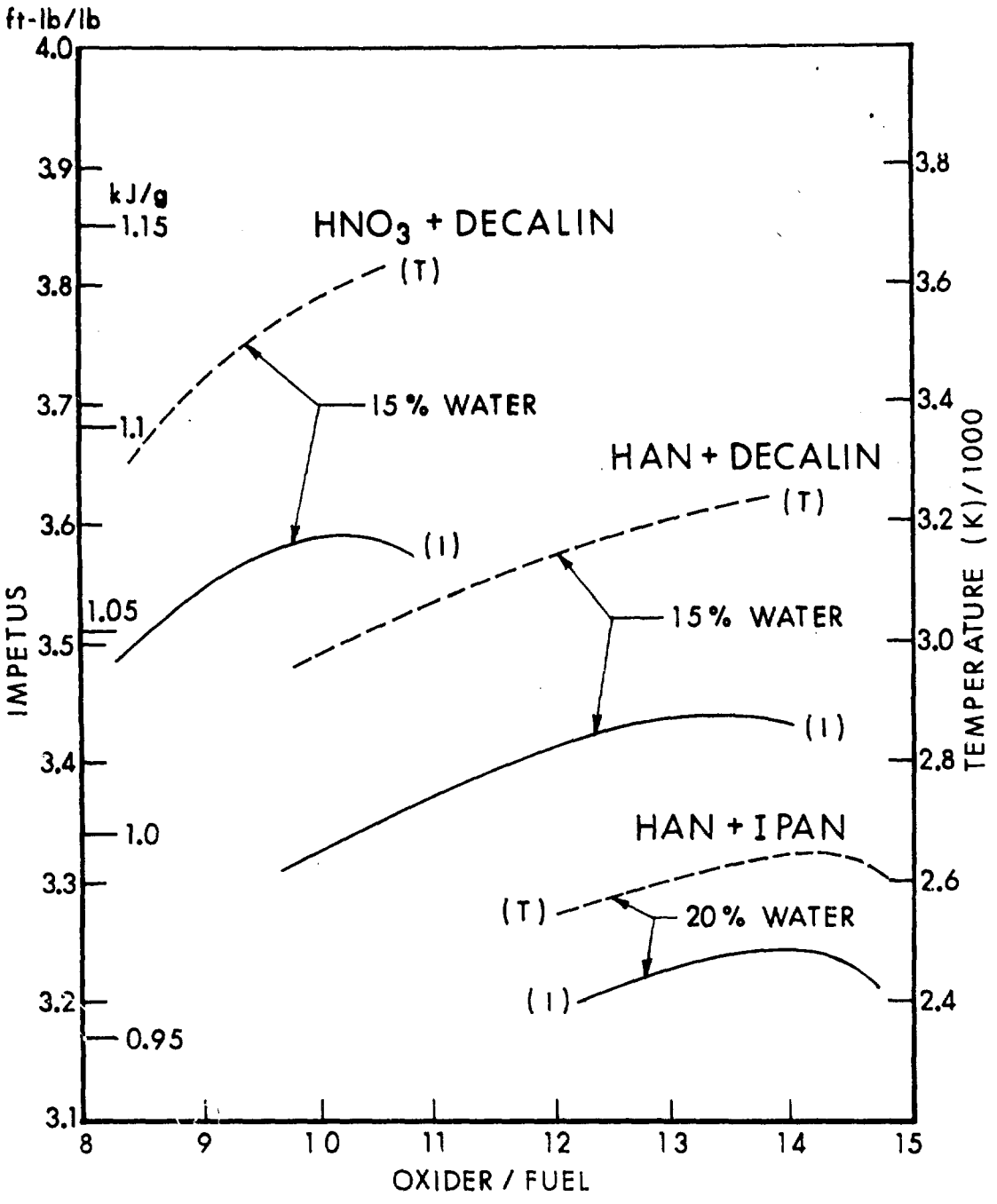

Figure 4. (U) Effect of Oxidizer/Fuel Ratio on Two Liquid Bipropellants.

(U) REFERENCES

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